NORTH ATLANTIC TREATY ORGANIZATION
ORGANISATION DU TRAITE DE L'ATLANTIQUE NORD

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MAS/165-MMS/4022
6 June 1983

To : See distribution below

Subject : STANAG 4022 MMS (EDITION 3) - SPECIFICATION FOR RDX (HEXOGENE) FOR DELIVERIES FROM ONE NATO NATION TO ANOTHER

References : a. MAS(ARMY)(62)641 dated 25 September 1962 (Edition 2)
b. AC/310-D/10 dated 1 April 1982

Enclosure : STANAG 4022(Edition 3)

1. The enclosed NATO Standardization Agreement which has been ratified by nations as reflected in page iii is promulgated herewith.

2. The references listed above are to be destroyed in accordance with local document destruction procedures.

3. AAP-4 should be amended to reflect the latest status of the STANAG.

ACTION BY NATIONAL STAFFS

4. National staffs are requested to examine page iii of the STANAG and, if they have not already done so, to advise the Defence Support Division of the International Staff, through their national delegation as appropriate of their intention regarding its ratification and implementation.

J.J.A. DOUCET
Major-General, CAAR
Chairman, MAS

DISTRIBUTION

Action: All members of the Army Board, MAS, except UK (for onward transmission to national authorities); UK - Director Standardization (STAN 2)

Information: SECGENNATO (DS Div); SACEUR; SACLANT; CINCHAN; CINCNORTH; CINCENT; CINCSOUTH; NAMSA

STANAG 4022
(Edition 3)

NORTH ATLANTIC TREATY ORGANIZATION
(NATO)

MILITARY AGENCY FOR STANDARDIZATION
(MAS)

STANDARDIZATION AGREEMENT

SUBJECT. SPECIFICATION FOR RDX (HEXOGENE) FOR DELIVERIES FROM ONE NATO NATION TO ANOTHER

Promulgated on 6 June 1983

J.J.A. DOUCET
Major-General, CAAR
Chairman, MAS

RECORD OF AMENDMENTS

No.	Reference/date of amendment	Date entered	Signature

EXPLANATORY NOTES

AGREEMENT

1. This NATO Standardization Agreement (STANAG) is promulgated by the Chairman MAS under the authority vested in him by the NATO Military Committee.

2. No departure may be made from the agreement without consultation with the tasking authority. Nations may propose changes at any time to the tasking authority where they will be processed in the same manner as the original agreement.

3. Ratifying nations have agreed that national orders, manuals and instructions implementing this STANAG will include a reference to the STANAG number for purposes of identification.

DEFINITIONS

4. Ratification is "The declaration by which a nation formally accepts the content of this Standardization Agreement"

5. Implementation is "The fulfilment by a nation of its obligations under this Standardization Agreement".

6. Reservation is "The stated qualification by a nation which describes that part of this Standardization Agreement which it cannot implement or can implement only with limitations".

RATIFICATION, IMPLEMENTATION AND RESERVATIONS

7. Page iii gives the details of ratification and implementation of this agreement. If no details are shown it signifies that the nation has not yet notified the tasking authority of its intentions. Page iv (and subsequent) gives details of reservations and proprietary rights that have been stated.

Agreed English/French Texts

NATO STANDARDIZATION AGREEMENT
(STANAG)

SPECIFICATION FOR RDX (HEXOGENE) FOR
DELIVERIES FROM ONE NATO NATION TO ANOTHER

ANNEXES : A. Melting Point Bath

Related documents : Nil

AIM

1. The aim of this agreement is to establish a common minimum specification for deliveries of RDX (Hexogene) from one NATO nation to another.

AGREEMENT

2. Participating nations agree that all RDX manufactured by NATO nations to be delivered to another NATO nation, except for particular use, must comply with the following minimum requirements:

PART 1 - PHYSICAL, CHEMICAL AND SAFETY CHARACTERISTICS

3. Composition. The RDX, Type A, produced by the nitric acid process shall consist essentially of cyclotrimethylene-trinitramine. RDX, Type B, produced by the acetic anhydride process, shall consist of cyclotrimethylene-trinitramine, with not more than 12% of cyclOtetra-methylene-tetranitramine.

4. Appearance and Granulation. The material shall be in the form of a white crystalline powder and shall conform to the granulation requirements specified in the contract.

5. Chemical Data:	Type A	Type B
a. Melting point:	min 200°C	min 190°C
b. Acetone insoluble:	max 0.05%	max 0.05%
c. Inorganic matter (Ash)	max 0.03%	max 0.03%
d. Acidity:		
(Quality):	(as nitric acid)	(as acetic acid)
Grade I (for detonators):	max 0.01%	max 0.01%
Grade I A (for other uses):	max 0.05%	max 0.02%

6. Safety Characteristics - Gritty Particles. Following extraction with acetone and sieving as described in paragraph 10, not more than 5 gritty particles per 50g sample shall be retained on a 0.0098 inch (0.25mm) aperture sieve and no gritty particles shall be retained on a 0.0165 inch (0.42mm) aperture sieve.

PART II - TESTING METHODS

7. General. When apparatus is not fully described in the following description normal laboratory procedures apply to the determination of the characteristics of the material.

8. Physical Tests - Sieve Test. To be conducted in accordance with the specification laid down in the contract.

9. Chemical Tests:

a. Melting Point The melting point of the RDX may be determined by either the Bloc Maquenne Method or the Capillary Tube Method. The method of test used shall be quoted on the test certificate.

(1) Bloc Maquenne Method:

(a) Apparatus:

1. To determine the melting point of RDX, an electrically heated Bloc Maquenne will be used, fitted with a mercury thermometer accurate to within 0.2°C over the whole thermometer scale.

2. Before use, the Block Maquenne will be examined to ensure:

(i) that the heating plate is not oxidised;
(ii) that the rate of heating of the area selected to determine the melting point is constant and that the temperature is uniform in this area.

(b) Product used:

1. In addition to the RDX, of which the melting point is to be ascertained, pure succinic acid, benzoylparachloranilide and dicyandiamide must be available.

2. All the products must be dry and pass through a sieve of aperture 0.0040 inch (0.10mm).

(c) Method:

1. Heat the Bloc Maquenne rapidly to about 175°C, then adjust the heating to obtain a rise of temperature of 1°C every three minutes.

2. For type B RDX as from 180°C, place on the plate of the Bloc Maquenne a few crystals of succinic acid (melting point provisionally stated to be 182.7°C) and renew them at least every thirty seconds until the melting point of the product is reached within 10 to 15 seconds. The moment this occurs read temperature T_1 on the thermometer.

3. Continue in this manner with the RDX to be tested and subsequently with the benzoylparachloranilide (melting point provisionally stated to be 192.4°C). Temperature T_2 and T_3 respectively are thus obtained on the thermometer.

4. For type A RDX adopt the same method but use benzoylparachloranilide (melting point provisionally stated to be 192.4°C) and dicyandiamide (melting point provisionally stated to be 205.9°C) as the reference products.

5. Calculate the differences:

for type B RDX: $T_1 - 182.7°C = a$
$T^1 - 192.4°C = b$
for type A RDX: $T^3 - 192.4°C = a$
$T^1_3 - 205.9°C = b$

a and b should be less than 1°C and (a - b) less than 0.2°C.

If the latter two conditions are not met the reason(s) must be found. It will be necessary to check the satisfactory operation of the Bloc Maquenne.

6. The melting point of the RDX tested is given by $T = T_2 - a$.

(2) Capillary Tube Method:

(a) Apparatus:

1. To determine the melting point of RDX the apparatus detailed below will be used:

(i) Thermometers, short range, short stem thermometers full immersion type, having a range of 195-205°C and 185-195°C for RDX type A and type B respectively, graduated at 0.10°C intervals.

(ii) Melting point bath, shown **at Annex A.** It consists of a tall form 800 ml beaker of resistance glass (A) closed with a chloroprene bung (B), through the centre of which passes a glass tube (C). The tube is closed with a chloroprene bung (D) through the centre of which passes the shaft of a mechanical stirrer (E), which shall be such that the direction of the flow of the liquid is downward. Four holes (F) in the tube allow circulation of the heating liquid.

The bung (B) has a hole to accommodate the thermometer (G) and, at a distance of about 1 cm small holes are bored on either side to accommodate melting point tubes (H).

The bath is filled with silicone oil or other suitable liquid to such a depth that the liquid circulates freely when the temperature of the bath is within 10-15°C of the melting point.

(iii) A suitable screen is necessary to protect the apparatus from draughts when in use.

(iv) Melting point tubes (H). Capillary tubes 150 mm long and 0.5 to 1.0 mm internal diameter.

(v) A second thermometer is necessary to indicate ambient temperature.

(b) Product used:

A portion of the sample shall be ground with caution to a fine powder and dried in a boiling water oven for 2 hours.

(c) Method:

1. Transfer to the dry capillary tube sufficient of the ground and dried sample to fill it to a depth of 1cm. Immerse the thermometer in the bath so that the bottom of the bulb is about 40 mm above the bottom of the beaker. Support a second thermometer, with its bulb as near as possible to the exposed stem of the main thermometer.

2. Run the mechanical stirrer so that the liquid flows downwards in the tube (C), and regulate the speed, about 600 revs/min. so that the surface of the liquid between (C) and (A) remains level.

3. Raise the temperature of the bath at a rate of 10°C per minute by means of a bunsen burner, taking care only to heat the base of the beaker, and protecting the flame and bath from draughts.

4. When the temperature of the bath is about 10°C below the expected melting point of the sample, insert the capillary tube containing sample in one of the small holes in bung (B), using a small paper "flag" for support if necessary, so that the bottom of the tube is level with the bottom of the thermometer bulb.

5. Reduce the heating rate so that temperature rises at 1 °C per minute. The materiel may slump and change colour as the melting point is approached. This appearance should not be confused with the first appearance of liquid.

6. Note the temperature T_1 at which liquid first appears and the temperature T_2 at which half the sample is molten. RDX decomposes as it melts and gas evolution is normally evident at the half melt stage.

7. Calculate the melting point as follows:

   Melting Point $^{o}C = T_2 + C_1 + C_2$

8. Where C_1 is the correction for thermometer bore errors at temperature T_2 and $C_2 = 0.00016N (T - t)$ where N = number of degrees divisions of exposed mercury column, T = indicated temperature in degree C and t = average ambient temperature indicated by a second thermometer whose bulb is near the mid-point of the exposed mercury column of thermometer G.

b. Acetone insoluble. Place a weighed portion of approximately 10 g of the dried sample in a 400 ml beaker and add 200 ml of acetone. Cover with a watch glass and heat on a steam bath until the RDX is dissolved. Filter the acetone solution through a tared filtering crucible, prepared by washing the filter with acetone igniting and weighing, and take care to transfer all the insoluble matter to the crucible. Wash the residue three times with acetone. Dry the crucible for 30 minutes in an oven at 105 °C, cool in a desiccator and weigh. Calculate the increase in weight as percentage of insoluble matter in the sample.

c. Inorganic matter. Ignite the material collected in the crucible as directed in paragraph b, above, cool and re-weigh. Calculate the increase in weight over the original tare weight as percentage of inorganic insoluble material.

d. Acidity:

(1) Weigh exactly 10 g ± 0.01 g of dry RDX and place it in a 500 ml extraction flask.

(2) Add 100 ml of acetone measured in a 100 ml test tube.

(3) Place a small condenser in the neck of the flask, heat over a water-bath or on a hot plate and shake by hand until the RDX is dissolved.

(4) Pour in slowly (20 to 30 seconds) 100 ml of distilled water measured in a 100 ml test tube and wait until the explosive settles (1 to 2 minutes).

(5) Add 8 to 10 drops of methyl red/methylene blue indicator (0.1 gm of methyl red and 0.05 g of methylene blue in 100 ml of 95% ethyl alocohol) and titrate at once, without filtering using 0.05 N sodium hydroxide. For this purpose use a 5 ml semi-microburette graduated in 1/50 ml, 1 ml corresponding to a length of 70 to 80 mm.

(6) Add the 0.05 N sodium hydroxide solution drop by drop to the flask, shaking until the indicator end-point is reached.

(7) Note the volume V_1 of solution used.

(8) Carry out a blank test simultaneously under identical conditions to those of the actual determination. For this purpose, place in a 500 ml or larger extraction flask 100 ml of acetone, 100 ml of distilled water and 8 to 10 drops of methyl red/methylene blue indicator. Titrate with 0.05 N sodium hydroxide solution. Note the volume of V_2 solution used.

The acidity of the RDX Type A expressed as percentage of nitric acid is given by:

$$0.0315\ (V_1 - V_2)\ f$$

The acidity of the RDX Type B expressed as percentage of acetic acid is given by:

$$0.03\ (V_1 - V_2)\ f$$

In both cases f is the correction factor for the normality of the sodium hydroxide solution.

10. Safety Tests - Gritty Particles. Transfer a weighed portion of approximately 50 g to a 0.0098 inch (0.25 mm) aperture sieve. Place the sieve in a Soxhlet apparatus or any other suitable extractor. Add sufficient acetone to the flask and extract on a steam bath until all the RDX is dissolved. Remove the sieve, count and examine the remaining particles. Brush the particles on a 0.0165 inch (0.42 mm) aperture sieve, count and examine any that are retained. Note if the particles are gritty, indicated by lack of uniformity of the material and the persistence of a scratching noise when the material is pressed and rubbed on a smooth glass slide with a smooth steel spatula

IMPLEMENTATION OF THE AGREEMENT

11 This STANAG is considered implemented when a nation has issued the necessary orders/instructions putting the contents of this agreement into effect.

A-1

MELTING POINT BATH - *BAIN DU POINT DE FUSION*

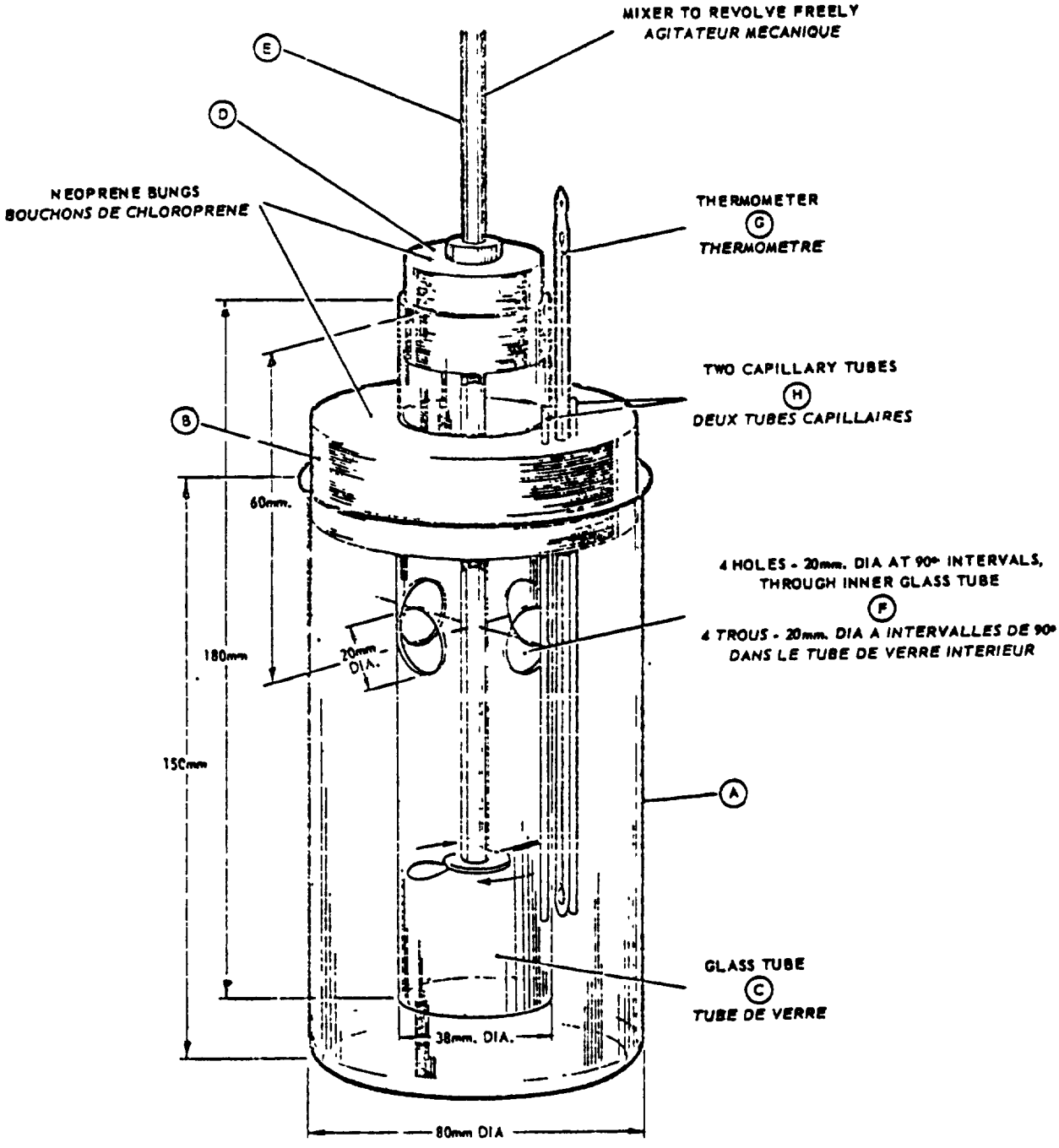

FIGURE 1

RATIFICATION AND IMPLEMENTATION DETAILS
STADE DE RATIFICATION ET DE MISE EN APPLICATION

NATION	NATIONAL RATIFICATION REFERENCE DE LA RATIFICATION NATIONALE	NATIONAL IMPLEMENTING DOCUMENT NATIONAL DE MISE EN APPLICATION	IMPLEMENTATION/MISE EN APPLICATION					
			FORECAST DATE DATE PREVUE			ACTUAL DATE DATE REELLE		
			NAVY MER	ARMY TERRE	AIR	NAVY MER	ARMY TERRE	AIR
BE	GS 5540 of/du 7.10.82						6.83	
CA								
DA								
FR								
GE	BMVg-Fü S IV 1 Az 03-51-60 of/du 30.8.82		10.83	10.83	10.83			
GR	5707/21/4873 of/du 23.11.82		1.83					
IT								
LU	GS 5540 of/du 7.10.82						6.83	
NL								
NO								
PO								
SP								
TU	4770(MAS)352 of/du 4.2.83							
UK	D/DSTAN/201/11/4022 of/du 26.8.82	DEF-STAN 07-23/1	83	83				
US								

*See reservation overleaf/
Voir réserve au verso

NATO EFFECTIVE DATE)
DATE D'ENTREE EN VIGUEUR OTAN)